Int. j. adv. multidisc. res. stud. 2025; 5(1):154-158

Received: 19-11-2024
Accepted: 29-12-2024

International Journal of Advanced Multidisciplinary Research and Studies

ISSN: 2583-049X

Characteristics of Edible Film from the Cassava Starch and Proteins

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Abstract
The production of edible film from cassava starch and proteins has been widely and dependently developed but still demonstrated the characteristics that do not meet international standards. So that needs to be developed edible film from both the material. This study aims to know the effect of a ratio variation of the cassava starch – proteins and the concentration of acetic acid to characteristics of edible film composites, determine a ratio of the cassava starch – proteins and the concentration of acetic acid solution to produce edible film with the best characteristics. This study used a Factorial Randomized Block Design with two factors. Factor I is a ratio of the cassava starch – proteins, which consists of 5 levels namely 100:0, 75:25, 50:50, 25:75, and 0: 100. The factor II is the concentration of acetic acid which consists of 5 levels, namely 0.5; 1.0; 1.5%. Each treatment combination was grouped into 3 blocks based on the time of the process of making edible film composites, so that there were 45 experimental units. The data obtained were analyzed of variant and followed by Duncan's multiple comparison tests. The variables observed were tensile strength, elongation at break and Young's modulus, swelling, the biodegradation time of edible film composites. The results showed that a ratio of the cassava starch – proteins, the concentration of acetic acid and its interaction had a very significant effect on tensile strength, elongation at break, Young's modulus, swelling and the degradation time of edible film composites. A ratio of the cassava starch-proteins= 75:25 with 1% acetic acid concentration produced the best edible film with the characteristics: Tensile strength of 9.84 MPa, elongation at break of 8.80%, Young's modulus of 225.42 MPa, swelling of 42.40% and the degradation time of 6.00 days.

Keywords: Edible Film, Ratio, Cassava Starch, Proteins, Acetic Acid Concentration

Introduction

The starch from cassava and proteins have great potential to be used as edible films. The production of starch and proteins based edible films independently has been widely developed, but still demonstrated poor characteristics. Darni and Utami (2010) [8] for instance, found that edible films made of starch have poor water resistance and mechanical strength. Romadloniyah (2012) [24] showed that adding of 1.5 ml sorbitol in the manufacture of edible film from cassava starch produced a tensile strength of 126.87 MPa and a percentage of elongation at break of 23.33%. Meanwhile Kumoro and Purbasari (2014) [19] stated that producing edible film composite using glycerol 2% (w/w) has characteristics: Young's modulus value of 40.5 MPa, tensile strength of 17 MPa and elongation at break of 38%. Harsojuwono and Arnata (2016) [14] that used the cassava starch (6%) combined with 1% glycerol resulting in a moisture content of 73.98%, elongation at a break of 18.75%, tensile strength of 9.30 MPa and Young's modulus of 50 MPa. Harsojuwono *et al* (2017) [15] made edible film with using the cassava starch by drying at 50^{o}C for 5 hours using automatic cabinet dryer with a debit air flow of 5 $\pm$ 0.1 m^3 / minute, resulting in tensile strength of 10.57 MPa, elongation at break of 15.95 %, Young's modulus of 66.29 MPa, swelling of 9.91% and degradation time of 7 days. Harsojuwono *et al*., (2018) [16] developed the research by treatment the gelatinization temperature at 75 $\pm$ 1^{o}C and pH 5, produce the cassava starch edible films with characteristics as follows: Tensile strength of 16.57 MPa, elongation at break of 10.32%, Young's modulus of 160.60 MPa, swelling of 129% and the degradation time of 7.33 days. Meanwhile Pradipta and Mawarani (2012) [22] made edible film from glucomannan with a stirring temperature of 80^{o}C and adding of 10 ml plasticizer, had a tensile strength of 0.035 MPa, swelling of 61.6% with a degradation time of 9 days. Those studies show that the characteristics of edible films still not meeting the International Plastic Standards (ASTM 5336).

Therefore, it is necessary to optimize the influential factors in making edible film such as mixtures polymer materials and using of acetic acid to produce a good quality edible film that meet the International Plastic Standards (Ban, 2006) [5].

The formation of edible film is basically influenced by many factors including a ratio of the mixtures polymer material and the concentration of acetic acid solution used as a regulator of acidity in gel formation (Henrique *et al*., 2007) [17]. Both of these things are critical on the composite characteristics formed (Harsojuwono, 2011) [11]. Dinda, *et al* (2014) [10] showed that carrageenan edible film and cassava starch in a ratio of 3: 2 using 0.5% sorbitol had a tensile strength of 49.2 MPa, elongation at break of 8.9%, Young's modulus of 1095 MPa and swelling of 35, 49%. Ariska and Suyatno (2015) [3] showed that edible film from banana hump starch and carrageenan in a 1: 2 ratio with glycerol as plasticizers had a tensile strength of 5.15 MPa, elongation at break of 14.25% and Young's modulus of 0.36 MPa. Meanwhile, Siswanti *et al* (2009) showed that glucomannan and cornstarch composites in a ratio of 15:85 had a thickness value of 0.18 mm, solubility of 50.58%, tensile strength of 1.49 MPa and elongation at a break of 30.56%. According to Maulana *et al*., (2016) [21] decrease in ratio of glucomannan and tapioca reduces the tensile strength and Young's modulus, but increased the elongation at break. However Abdurrozag (2016) [1] showed that the mixture ratio of 30% proteins, 70% starch with 25% glycerol had characteristics with a tensile strength of 9.3 MPa, elongation at break of 44.68%, swelling of 136.73% and the degradation time of 20.5 days. Primaningrum and Sari, (2014) [23] produced glucomannan and chitosan composites in a 1: 3 ratio using 0.5% acetic acid solution and 3% glycerol had a tensile strength of 2.14 MPa, Young's modulus value of 9.52 MPa, and elongation at break 22.5%, swelling of 41.67% with a degradation time of 6 days. Dewi (2015) [9] produced an edible film composite of cassava skin starch and chitosan in a 4: 1 ratio using 1% acetic acid solution and 1.5% glycerol produced a tensile strength of 0.39 MPa, elongation at a break of 44.06%, Young's modulus of 0.08 MPa and the degradation time of 7 days. Al Hasan and Norziah (2012) explain that the formation of composite films from a mixture of starch and gelatin will improve its mechanical properties compared to the formation of films from one of the material. The above finding shows that a ratio of the mixture of polymer materials and the concentration of acetic acids influenced the characteristics of edible film composites. The optimum value of cassava starch– proteins ratio and acetic acid concentration in the manufacture of edible film has not been identified. Thus, it is necessary to explore the mixtures ratio of the cassava starch – proteins and the concentration of acetic acid solution that can improve and enahnce the edible films composites characteristics in accordance with the International Plastic Standards. The purpose of this study was to investigate the effect of the cassava starch – proteins ratio and acetic acid concentration to the characteristics of edible film composites.

Materials and methods

Material: The materials used in this study were the cassava starch from Indo Food Chem., pure proteins from CV Nura Jaya, acetic acid, glycerol, and aquadest from Bratha Chem.

Research design: Using factorial randomized block design. The first factor is a ratio of the cassava starch- proteins, consisting of 5 levels, namely 100:0, 75:25, 50:50, 25:75, and 0: 100. The second factor is the concentration of acetic acid which consists of 3 levels, namely 0.5; 1.0; 1.5%. Each treatment combination is grouped into 3 based on the time of the edible film manufacturing process, with total of 45 experimental units.

Making of edible film

Considering 6 g of a mixture of cassava starch and proteins with a ratio according to treatment, then added 93 g of acetic acid solution with a concentration according to treatment then stirred for 10 minutes with a spatula in beaker glass, then added with plasticizer glycerol 1 g. following a 10-minutes stirring process for homogenization. Next, the mixture is heated and stirred on the water bath at 75 $\pm$ 1ºC until it formed a gel. The formed gel is then printed on Teflon with a diameter of 20 cm. After that, it is dried in an oven at 50ºC for 5 hours. The edible film composite formed was cooled for 24 hours at room temperature (27$\pm$1ºC). Next, the edible film is removed from the Teflon and is ready to be analyzed for its characteristics.

Observation variables

Tensile strength, elongation at break, Young's modulus (ASTM D638), swelling (Harsojuwono, 2016) [14], degradation time / biodegradation (ISO 17556).

Data analysis

The data obtained were analyzed of variant and followed by Duncan's multiple comparison tests. The program used for data analysis was SPSS 25.

Results and discussion

Tensile strength, elongation at break and Young's modulus

Analysis of variant shows that a ratio of the cassava starch – proteins and acetic acid concentration and their interactions have a very significant effect on tensile strength, elongation at break and Young's Modulus of edible film composites. The mean values of tensile strength, elongation at break and Young's Modulus of edible film ranged from 4.36-9.84 MPa, 8.80-19.56% and 118.74-225.60 MPa, respectively, as shown in Table 1.

The Table 1 shows that the highest mean of tensile strength with value of 9.84 MPa owned by the composite with a ratio of the cassava starch – proteins= 75:25 with addition of 1% acetic acid. The lowest mean of tensile strength values were from edible film with a ratio of the cassava starch – proteins= 0:100 added with 1.5% acetic acid concentration. Getting lower a ratio of the cassava starch – proteins, initially increasing tensile strength but after achieving the optimum ratio, the decrease in the ratio of cassava starch– proteins caused a decrease in tensile strength of edible film composite. This is in accordance with Jian *et al* (2016) [18] who explained that the greater increase in glucomannan in the Tilapia myofibrillar protein caused a decrease in the strength of the gel from the edible film composite. Meanwhile, an increase in the concentration of acetic acid also decreases the tensile strength value after optimum conditions. This is in accordance with result of the research Yang *et al* (2013) [29] who stated that the use of acidic pH or high acidity will cause degradation during the gelatinization process which has an impact on tensile strength. In this

study, the highest mean tensile strength was owned by edible film with a ratio of the cassava starch – proteins= 75: 25 at 1% acetic acid concentration of 9.8 MPa. This value is higher than edible film from the mixture proteins, starch and glycerol with ratio 30:70:25 that has tensile strength values of 9.3 MPa (Abdurrozag, 2016) [1]. This edible film has tensile strength value is higher than edible film of glucomannan and corn starch with tensile strength values of 1.49MPa (Siswanti, *et al*., 2013) [27] and either edible film from glucomannan-based from research of Maulana *et al* (2016) [21] which has a tensile strength value of 0.67 MPa.

Table 1 also shows that the high mean elongation at break is found in edible film with a ratio of cassava starch – protein = 0:100 and acetic acid concentration of 1 and 1.5% with a value of 19.03-19.56%. Meanwhile, the lowest mean of elongation at break is shown by edible film with a ratio of the cassava starch – proteins= 75: 25 at 1% acetic acid concentration with value of 8.80%. This value shows that the mean of elongation at break is lower than elongation at break of the cassava starch edible film which has the value is 10.32% (Harsojuwono *et al*, 2018) [16] but still higher than elongation at break of the breadfruit and chitosan starch composite which has the value of 6.00% (Setiani *et al*., 2016) [26]. The value of elongation at break from an edible film composite of the cassava starch – proteins has met international plastic standards (ASTM5336) which stipulate that elongation at break is less than 500% for PCL plastics from England, as well as PLA plastic standards from Japan that set maximum of the elongation at break of 9%.

Table 1 also shows the highest mean Young's Modulus owned by edible film with a ratio of the cassava starch – proteins= 75:25 at 1% acetic acid concentration with a value of 225.42 MPa. The lowest mean of Young's Modulus is also owned by edible film with a ratio of the cassava starch – proteins= 0:100 at acetic acid concentration of 1.5%. The edible film with a ratio of the cassava starch – proteins= 75: 25 at 1% acetic acid concentration had the higher Young's Modulus than the composites of glucomannan and chitosan in a ratio of 1: 3 at 0.5% acetic acid concentration with Young's Modulus values 9.52 MPa (Primaningrum *et al*., 2014) [23], as well as the composites of cassava skin starch and chitosan in a 4: 1 ratio at 1% acetic acid concentration with Young's modulus value of 0.08 MPa (Dewi, 2015) [9]. According to Leuangsukrerk *et al* (2014) [20] an increase in the ratio of glucomannan and whey protein causes an increase in Young's modulus, and tensile strength but decreases the transparency of the film composites.

Table 1: The mean values of tensile strength, elongation at break and Young's modulus of edible film in ratio variations of the cassava starch – proteins and acetic acid concentration

Ratio of the cassava starch- proteins and acetic acid concentration	Mean of tensile strength (MPa)	Mean of elongation at break (%)	Mean of Young's modulus (MPa)
100 : 0 ; 0.5%	8.70^{b}	14.70^{c}	186.13^{c}
100 : 0 ; 1%	8.93^{b}	11.32^{de}	208.37^{b}
100 : 0 ; 1.5%	8.63^{b}	10.03^{a}	195.99^{bc}
75 : 25 ; 0.5%	8.70^{b}	10.70b^{c}	205.14^{b}
75 : 25 ; 1%	9.84^{a}	8.80^{f}	225.42^{a}
75 : 25 ; 1.5%	8.60^{b}	9.99^{e}	200.65^{b}
50 : 50 ; 0.5%	8.21^{bc}	10.32^{de}	197.23^{bc}
50 : 50 ; 1%	7.72^{c}	10.93^{de}	189.30^{c}
50 : 50 ; 1.5%	7.36^{c}	11.56^{d}	182.71^{c}
25 : 75 ; 0.5%	6.41^{cd}	12.32^{d}	173.46^{cd}
25 : 75 ; 1%	6.32^{d}	15.93^{bc}	170.05^{d}
25 : 75 ; 1.5%	6.06^{d}	16.56^{b}	168.99^{d}
0 : 100 ; 0.5%	5.21^{d}	17.96^{b}	148.50^{e}
0 : 100 ; 1%	5.32^{d}	19.03^{a}	128.26^{f}
0 : 100 ; 1.5%	4.36^{e}	19.56^{a}	118.74^{g}

Description: The same notation in the same column does not show a significant difference at the 5% significance level

Swelling and biodegradability

Analysis of variant showed that a ratio of the cassava starch – proteins and acetic acid concentration and their interactions significantly affect to the mean of swelling and degradation times. The mean of swelling ranged from 42.40 – 121.57%, while the degradation time ranged from 3.33 to 7.33 days.

Table 2 shows that the highest mean swelling (121.57%) is possessed by edible film with a ratio of cassava starch– protein = 0:100 at an acetic acid concentration of 1.5%. Meanwhile, edible film with a ratio of cassava starch– protein = 75: 25 at acetic acid concentration of 1.0% had the lowest mean swelling (42.40%). There is a tendency that the lower the ratio of the cassava starches to the protein, the higher the swelling of edible film composites. This seems to be related to the ability of protein to absorb and retain water in the gel which reaches more than 300% of the weight of the dry matter. This is in accordance with the study by Leuangsukrerk *et al* (2014) [20] which showed that the higher glucomannan ratio to whey protein caused a decrease in integrity but increased swelling from edible film composites. According to Maulana *et al* (2016) [21], the lower ratio of the tapioca and protein causes an increase in swelling of biopolymer composites. Further explained that the ratio of tapioca and protein = 5: 5 causes an increase in swelling reaching 33.12%. Meanwhile, according to Cheng *et al* (2006) [6], this is related to the extensive interaction between plasticizers and main matrix which affects the active (OH) side, thus affecting the ability to absorb water or swelling.

Table 2: Swelling and the degradation time of edible film in variations in a ratio of the cassava starch – proteins and acetic acid concentration

Ratio of the cassava starch-protein and acetic acid concentration	Mean of swelling (%)	Mean of degradation time (day)
100 : 0 ; 0.5%	69.12^{d}	7.33^{a}
100 : 0 ; 1%	62.00^{de}	7.33^{a}
100 : 0 ; 1.5%	59.62^{d}	7.00^{a}
75 : 25 ; 0.5%	51.12^{f}	6.33^{ab}
75 : 25 ; 1%	42.40^{g}	6.00^{ab}
75 : 25 ; 1.5%	50.59^{f}	6.33^{ab}
50 : 50 ; 0.5%	61.33^{e}	5.33^{b}
50 : 50 ; 1%	65.67^{de}	5.33^{b}
50 : 50 ; 1.5%	66.79^{d}	5.33^{b}
25 : 75 ; 0.5%	67.25^{d}	4.00^{bc}
25 : 75 ; 1%	79.37^{cd}	4.33^{bc}
25 : 75 ; 1.5%	82.47^{c}	5.67^{b}
0 : 100 ; 0.5%	102.35^{b}	3.33^{e}
0 : 100 ; 1%	109.65^{b}	3.33^{c}
0 : 100 ; 1.5%	121.57^{a}	3.33^{c}

Description: The same notation in the same column does not show a significant difference at the 5% significance level

Table 2 also shows that the mean degradation time of edible film with a ratio of cassava starch – protein = 100:0 with acetic acid concentrations of 0.5, 1.0, and 1.5% has a long time. This is not significantly different from the time degradation of edible film with a ratio of cassava starch - protein = 75:25 at acetic acid concentrations of 0.5, 1.0, 1.5%. While the average degradation time of edible film with a ratio of cassava starch - protein = 0:100 at an acetic acid concentration of 0.5, 1.0, and 1.5% has a short time, which is significantly different from the degradation time of other edible films. There is a tendency that the smaller a ratio of the cassava starch to the protein causes shorter the degradation time. According to Torres *et al* (2011) [28], all starch-based films will be decomposed in three stages of the process, which are weight loss of up to 30% due to glycerol washing, loss of up to 90% due to biological activity, loss of up to 95% due to further biodegradation which causes reduced the mechanics properties. Further explained that rate of the weight loss of starch-based films is higher than rate of the weight loss of cellulose-based films. The edible film of cassava starch and protein have a mean of degradation time of 3.33 - 7.33 days. This is shorter than the degradation time of a mixture of 30% glucomannan and 70% starch which has a degradation time of 20.5 days (Abdurrozag, 2016) [11]. The degradation time of other composites that entered the degradation time span of edible film from cassava starch– proteins (3.33-7.33 days) was a bio-composite of research results by Primaningrum and Sari, (2014) [23]. Primaningrum and Sari (2014) [23] showed that edible film of the glucomannan and chitosan with a 1: 3 ratio using 0.5% acetic acid solution had a 6-day degradation time. Likewise, the results of Dewi's research (2015) [9] showed that the degradation time of composites edible film the cassava skin starch and chitosan in a 4: 1 ratio with 1% acetic acid solution was 7 days. This degradation time is still shorter than the standard PLA plastic from Japan and the PCL from England which sets 60 days.

Conclusion

The conclusion of this study were a ratio of the cassava starch – proteins, the concentration of acetic acid and its interaction had a very significant effect on tensile strength, elongation at break, Young's modulus, swelling and the degradation time of edible film composites. The best edible film obtained at a ratio of the cassava starch – proteins = 75:25 with 1% acetic acid concentration that had characteristics: tensile strength of 9.84 MPa, elongation at break of 8.80%, Young's modulus of 225.42 MPa, swelling of 42.40% and the degradation time of 6.00 days.

Acknowledgement

We would like to thank Udayana University for providing research funding and facilitating the publication of this scientific article.

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